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IS 11788 (1986): 2-amino-5-diethylaminotoluene
Hydrochloride, Photographic Grade [CHD 5: Electroplating
Chemicals and Photographic Materials]



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“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

SPECIFICATION FOR
2-AMINO-5-DIETHYLAMINOTOLUENE
HYDROCHLORIDE, PHOTOGRAPHIC GRADE

UDC 547.553.2 : 771.7



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INDIAN STANDARDS INSTITUTION

MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR 2-AMINO-5-DIETHYLAMINOTOLUENE HYDROCHLORIDE, PHOTOGRAPHIC GRADE

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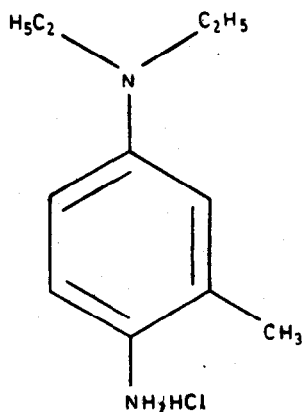
Indian Standard

SPECIFICATION FOR 2-AMINO-5-DIETHYLAMINOTOLUENE HYDROCHLORIDE, PHOTOGRAPHIC GRADE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 29 August 1986, after the draft finalized by the Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

0.2 2-Amino-5-diethylaminotoluene hydrochloride (also called 4-amino-3-methyl-N, N-diethylaniline hydrochloride) is used as a photographic colour developer. It is also known by trade names like colour developer-2, purechrome-2, CD-2, Activol-2 and Tolochrome. It has the following structural formula.



(Molecular Mass 214.45)

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard

*Rules for rounding off numerical values (revised).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for 2-amino-5-diethylaminotoluene hydrochloride used as the basic constituent of developing solutions for treatment of multilayer light sensitive materials of colour photography and cinematography.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of white to light tan, free flowing crystals, free from aggregates or large crystals and shall consist essentially of 2-amino-5-diethylaminotoluene hydrochloride.

2.2 Solution in Water — A freshly prepared aqueous solution of the material containing 5 g of the sample in 100 ml of water should be clear and free from sediment other than a slight flocculence.

2.3 The material shall comply with the requirements prescribed in Table 1 when tested according to the methods given in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR 2-AMINO-5-DIETHYLAMINOTOLUENE HYDROCHLORIDE

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL No. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Free base, percent by mass	83.0 ± 0.5	A-2
ii)	Hydrochloric acid, percent by mass	17.0 ± 0.25	A-3
iii)	Ash, percent by mass, <i>Max</i>	0.1	A-4
iv)	Iron (as Fe), percent by mass, <i>Max</i>	0.001	A-5
v)	Heavy metals (as Pb), percent by mass, <i>Max</i>	0.001	A-6
vi)	pH of 5 percent aqueous solution	5.0-5.5	A-7
vii)	Volatile matter at 70°C, percent by mass, <i>Max</i>	0.2	A-8
viii)	Identity test	To pass test	A-9

3. PACKING, MARKING AND SAFETY PRECAUTIONS

3.1 Packing — The material shall be packed in suitable, dark coloured, air-tight bottles or as agreed to between the purchaser and the supplier.

3.2 Marking — The container shall be securely closed and legibly and indelibly marked with the following information:

- a) Name and grade (photographic) of the material;
- b) Mass of the material;
- c) Date of manufacture;
- d) Manufacturer's name and/or his recognized trade-mark, if any; and
- e) Lot number in code or otherwise to enable the batch of manufacture to be traced from records.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

3.3 Safety Precautions — 2-Amino-5-diethylaminotoluene hydrochloride has a pronounced general toxic action and shows particular effect on liver and central nervous system. On skin contact, it may cause eczema.

3.3.1 Work with 2-amino-5-diethylaminotoluene hydrochloride should be done only with protective clothing. The place should be provided with exhaust and ventilation systems.

4. SAMPLING

4.1 Representative samples of the material shall be drawn and their conformity to the standard determined as prescribed in Appendix B.

APPENDIX A

(Clause 2.3, and Table 1)

METHODS OF TEST FOR 2-AMINO-5-DIETHYLAMINO-TOLUENE HYDROCHLORIDE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Specification for water for general laboratory use (second revision).

A-2. DETERMINATION OF FREE BASE

A-2.0 General — Using standard ceric sulphate solution, the free base content is found by volumetric method.

A-2.1 Reagents

A-2.1.1 Sulphuric Acid — 1 N.

A-2.1.2 Ferroin Indicator — See 3.3.7.1 of IS : 2263-1979*.

A-2.1.3 Standard Ceric Sulphate Solution — 0.05 N.

A-2.2 Procedure — Weigh accurately about 2.5 g of sample into 500-ml volumetric flask and dissolve it in 1 N sulphuric acid (H_2SO_4). Make up to the mark with 1 N sulphuric acid.

A-2.2.1 In a one-litre conical flask, take 100 ml of dilute sulphuric acid and 200 ml of water and add 10 to 12 drops of ferroin indicator. Titrate with ceric sulphate solution to a pale blue end point. Record the blank titre value V_1 . Pipette 25 ml of the sample solution prepared in A-2.2 into the conical flask and continue titration without refilling the burette until a loss of orange colour is observed momentarily. Reduce the rate of addition as end point is approached, so that one drop is added every 3 to 4 seconds near the end point. The end point is of a greenish-yellow colour which does not change with the addition of one more drop. Record the net titre value V .

A-2.3 Calculation

$$\text{Free base} = \frac{(V - V_1) \times F \times 89.14}{10 \times M}$$

where

V = net titre value,

V_1 = blank titre value,

F = factor of 0.05 N ceric sulphate solution, and

M = mass in g of the sample taken for the test.

A-3. DETERMINATION OF HYDROCHLORIC ACID

A-3.0 General — Hydrochloric acid is determined gravimetrically by precipitating with silver nitrate.

*Methods of preparation of indicator solutions for volumetric analysis (*first revision*).

A-3.1 Apparatus

A-3.1.1 Sintered Glass Crucible (Porosity G-3)

A-3.1.2 Electric Oven — preferably with automatic temperature control device.

A-3.2 Reagents

A-3.2.1 Sulphuric Acid — 1 N.

A-3.2.2 Concentrated Nitric Acid — with specific gravity 1.42.

A-3.2.3 Dilute Nitric Acid — 0.05 N (approx).

A-3.2.4 Silver Nitrate Solution — 0.1 N (approx).

A-3.3 Procedure — Weigh accurately 0.4 ± 0.01 g of the sample into 250-ml beaker covered with thick brown paper. Add 100 ml of 1 N sulphuric acid and stir until the sample has dissolved. Then add 0.5 ml of concentrated nitric acid and warm the solution to 60-70°C. Add freshly prepared 0.1 N silver nitrate solution slowly, using a graduated pipette with constant stirring, until the precipitation is completed, the precipitation is to be carried out in subdued light. Heat to boiling, while stirring constantly and maintain it at this temperature until the precipitates coagulate and the supernatant liquid is clear. Set the beaker aside in the dark for one hour. Filter and collect the precipitates in a tared sintered glass crucible covered with thick brown paper. Wash the precipitates in the crucible with dilute nitric acid added in small portions until washing gives no positive test for silver nitrate. Remove the brown paper cover and dry the crucible and its contents in an oven at 105°C for about two hours. Allow to cool in desiccator protected from light and weigh to get constant mass.

A-3.4 Calculation

$$\text{Hydrochloric acid, percent by mass} = \frac{M_1 \times 0.2546 \times 100}{M}$$

where

M = mass in g of sample taken, and

M_1 = mass in g of residue obtained.

A-4. DETERMINATION OF ASH

A-4.0 General — A known quantity of the material is ignited at 600°C and ash is cooled and weighed.

A-4.1 Apparatus

A-4.1.1 Silica Crucible

A-4.1.2 Furnace — preferably with automatic temperature control device.

A-4.2 Procedure — Weigh accurately 5.0 ± 0.1 g of the material in a tared silica crucible, moist it with 4 to 8 drops of concentrated sulphuric acid. Incinerate and then ignite at dull red-heat (approximately 600°C) to complete the ignition. Cool in a desiccator and weigh to get constant weight. Preserve the ash for iron and heavy metals test.

A-4.3 Calculation

$$\text{Ash, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g of the residue obtained, and

M_2 = mass in g of the material taken for the test.

A-5. DETERMINATION OF IRON

A-5.0 General — Two methods have been prescribed for the determination of iron, namely, *o*-phenanthroline method and thioglycollic acid method. However, in case of dispute, the *o*-phenanthroline method shall be the referee method.

A-5.1 *o*-Phenanthroline Method

A-5.1.1 Reagents

A-5.1.1.1 Acetate buffer, pH 5 — Add 23 g of anhydrous sodium acetate to 58 ml of 2 molar acetic acid and dilute to one litre with water. Adjust the final pH of the solution to 5.0 ± 0.1 with acetic acid or 10 percent sodium hydroxide solution.

A-5.1.1.2 Hydroxylamine hydrochloride — 10 percent aqueous solution (*m/v*).

A-5.1.1.3 *o*-Phenanthroline (1 : 10-phenanthroline) — 0.1 percent solution (*m/v*).

A-5.1.1.4 Standard iron solution — Dissolve 7.02 g of crystalline ferrous ammonium sulphate [$\text{Fe} (\text{NH}_4)_2 (\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$] in about 300 ml of water containing 2 ml of concentrated sulphuric acid and dilute to 1 000 ml with water. Further dilute 1 ml to 1 000 ml freshly when required. One millilitre of the diluted solution contains 0.001 mg of iron (as Fe).

A-5.1.2 Procedure

A-5.1.2.1 Add 5 ml of concentrated hydrochloric acid to the residue obtained from the determination of ash (*see A-4*), and evaporate to dryness. Dissolve the residue in 15 ml of dilute hydrochloric acid and warm on a steam-bath for 10 minutes. Cool, dilute to 50 ml with water in a volumetric flask and mix thoroughly. Use 20 ml of this solution for the determination of iron and preserve the remaining solution for heavy metals test.

A-5.1.2.2 Thoroughly mix equal parts of *o*-phenanthroline, hydroxylamine hydrochloride and acetate buffer of pH 5. Add 5 ml of this mixture to 20 ml portions of both the standard iron solution and the sample solution. Dilute to 50 ml with water and mix well.

A-5.1.2.3 The material shall be taken to have not exceeded the limit prescribed for iron, if the colour produced is not deeper than that produced by the standard iron solution.

A-5.2 Thioglycollic Acid Method

A-5.2.0 General — Add concentrated hydrochloric acid to the residue obtained from the determination of ash, evaporate to dryness and finally dissolve in dilute hydrochloric acid. Add thioglycollic acid, make the solution alkaline and compare the colour produced with that produced by standard iron solution under identical conditions.

A-5.2.1 Apparatus

A-5.2.1.1 Nessler cylinders — 50 ml capacity.

A-5.2.2 Reagents

A-5.2.2.1 Dilute hydrochloric acid — 10 percent (*v/v*).

A-5.2.2.2 Thioglycollic acid — 89 percent purity, *Min.*

A-5.2.2.3 Dilute ammonium hydroxide — 1 : 2.

A-5.2.2.4 Citric acid solution — 25 percent (*m/v*).

A-5.2.3 Procedure

A-5.2.3.1 Transfer a 20 ml aliquot of the sample solution from **A-5.1.2.1** to a Nessler cylinder and dilute to about 30 ml. Into the second Nessler cylinder containing 5 ml of dilute hydrochloric acid, pipette 20 ml of standard iron solution and dilute to about 30 ml with water. To both cylinders, add 2 ml of citric acid solution and 2 to 3 drops of thioglycollic acid. Mix, make just alkaline with ammonium hydroxide and dilute to 50 ml.

A-5.2.3.2 The material shall be taken to have not exceeded the limit prescribed for iron, if the colour produced is not deeper than that produced by the standard iron solution.

A-6. DETERMINATION OF HEAVY METALS

A-6.0 General — The colour produced by heavy metals in the sample on treating with hydrogen sulphide is compared with that produced with standard lead solution under identical conditions.

A-6.1 Reagents

A-6.1.1 Dilute Ammonium Hydroxide — 10 percent (*m/m*).

A-6.1.2 Dilute Hydrochloric Acid — approximately 0.1 N.

A-6.1.3 *p*-Nitrophenol Indicator — 0.25 percent aqueous solution (*m/v*).

A-6.1.4 Hydrogen Sulphide — saturated aqueous solution.

A-6.1.5 Standard Lead Solution — Dissolve 0.64 g of lead nitrate in water; add one millilitre of concentrated nitric acid and dilute to 1 000 ml with water. Further dilute 10 to 1 000 ml freshly when required. One millilitre of the resulting solution contains 0.004 mg of Pb.

A-6.2 Procedure

A-6.2.1 To the 20 ml portion of the sample solution (*see A-5.1.2.1*) in a Nessler cylinder add a few drops of *p*-nitrophenol indicator and then add ammonium hydroxide dropwise, until the solution turns yellow. Add dilute hydrochloric acid dropwise, until the solution becomes colourless again and then add 0.5 ml in excess. Add 5 ml of hydrogen sulphide solution and dilute to 50 ml.

A-6.2.2 At the same time transfer 5 ml of standard lead solution into another Nessler cylinder and render it neutral to *p*-nitrophenol indicator by using dilute ammonium hydroxide and dilute hydrochloric acid as described in **A-6.2.1**. Add 0.5 ml of dilute hydrochloric acid in excess and 5 ml of hydrogen sulphide solution. Dilute to 50 ml.

A-6.2.3 The material shall be considered to have passed the requirement of the test if the colour produced in the sample solution is not more intense than that produced in the standard solution.

A-7. pH OF THE SOLUTION

A-7.0 General — The pH of a 5 percent solution of the material is determined by using a suitable pH meter.

A-7.1 Apparatus

A-7.1.1 pH Meter — with glass and calomel electrodes.

A-7.2 Procedure — Prepare a 5 percent solution of the material in a 100-ml beaker, using freshly boiled and cooled water. Measure the pH of the solution using a pH meter.

A-8. DETERMINATION OF VOLATILE MATTER

A-8.0 General — A known quantity of the material is dried at 70°C and loss in mass is taken as volatile matter.

A-8.1 Apparatus

A-8.1.1 Glass-Stoppered Shallow Weighing Bottle

A-8.1.2 Oven — preferably with automatic temperature control device.

A-8.1.3 Desiccator — See IS : 6128-1971*.

A-8.2 Procedure — Weigh accurately 3 ± 0.1 g of the material in the weighing bottle and dry in the oven at 70°C for about 8 hours. Cool in the desiccator and weigh.

A-8.3 Calculation

$$\text{Volatile matter, percent by mass} = \frac{(M - M_1)}{M} \times 100$$

where

M = mass in g of the material taken for the test, and

M_1 = mass in g of the material obtained on drying.

A-9. IDENTITY TEST

A-9.0 Two methods namely Infrared and Thin Layer Chromatographic (TLC) have been prescribed. However, in case of dispute, the infrared method shall be the referee method.

A-9.1 Infrared Test

A-9.1.0 Outline of the Method — Using an infrared spectrophotometer the spectrum of the material is obtained which is compared with the reference spectrum of the known sample.

A-9.1.1 Apparatus — Infrared spectrophotometer equipped for 2.5 to 15 micron region and a provision to use pressed potassium bromide pellets.

A-9.1.2 Procedure — Grind about 1 g of the sample to a homogeneous fine powder. Prepare a 1 percent (m/m) mixture of the sample in potassium bromide. Grind thoroughly to about 200 mesh. Prepare a pressed pellet of the mixture to contain 0.2 to 0.3 g of the material per square cm. Record the infrared spectrum from 2 to 15 micron. Compare with the reference spectrum as given in Fig. 1.

*Specification for desiccators.

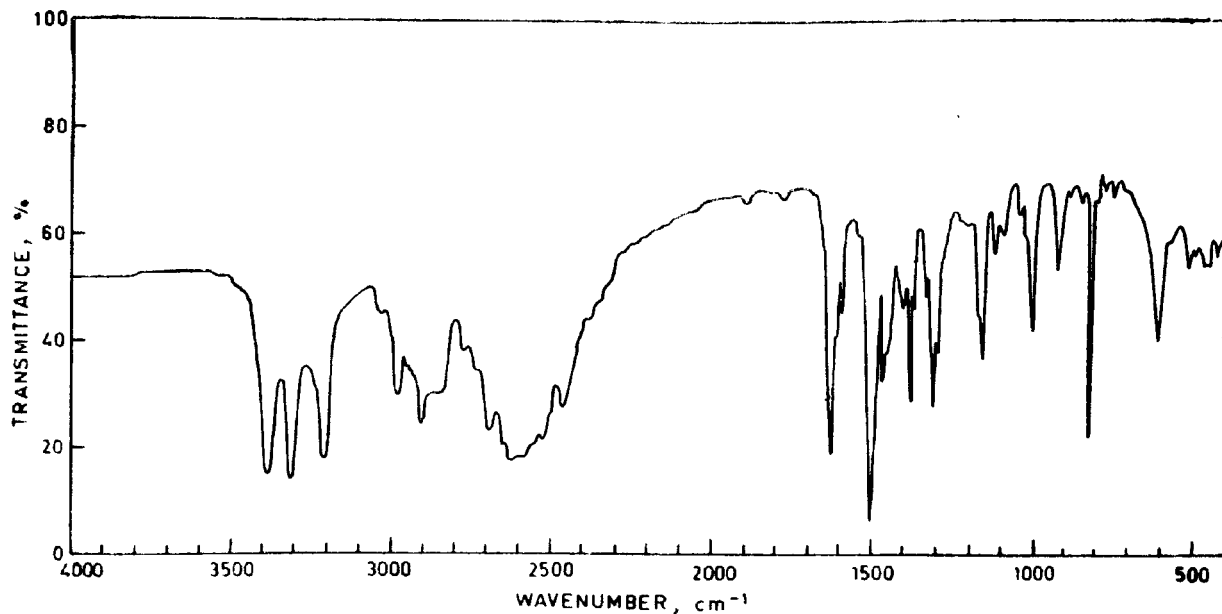


FIG. 1 REFERENCE IR SPECTRUM OF 2-AMINO-5-DIETHYLAMINOTOLUENE
HYDROCHLORIDE, PHOTOGRAPHIC GRADE

A-9.2 TLC Method

A-9.2.0 Outline of the Method — The TLC identity test is carried out by spotting both the test sample solution and the reference sample solution simultaneously and comparing the spots against each other after development.

A-9.2.1 Apparatus

A-9.2.1.1 TLC chamber

A-9.2.1.2 Micropipette of 10 microlitre capacity

A-9.2.1.3 Glass plates coated with silica gel G (10×20 cm)

A-9.2.1.4 Ammonia chamber

A-9.2.2 Reagents

A-9.2.2.1 Methanol — AR grade.

A-9.2.2.2 Benzene

A-9.2.3 Eluent — Benzene : Methanol (90 : 10 v/v).

A-9.2.4 Spray Reagent — A mixture composed of the following Reagents A and B:

Reagent A — 4 percent (m/v) ferric chloride (FeCl_3) solution in water.

Reagent B — 2 percent (m/v) potassium ferricyanide solution in water.

A-9.2.4.1 Mix reagents A and B in 2 : 1 proportion and dilute to three times with water.

A-9.2.5 Procedure — Dissolve 0.2 g of sample (on 100 percent basis) in 10 ml of 1 : 1 methanol—water mixture in a volumetric flask by warming. Prepare a standard solution in the similar matter. Spot 10 microlitre of both standard and sample solutions on a silica gel G plate and dry the spots thoroughly. Neutralize the spots by keeping the plate in ammonia chamber for about 10 minutes. Put the plate for elution in the chromatography tank containing benzene : methanol (90 : 10) as eluent. Allow to run the plate up to a length of 15 cm, dry and spray with spraying reagent. Evaluate visually.

A-9.2.5.1 Compare the intensity of the colour of the spot of the test material with that of the reference sample. Both should have a R_f value of 0.76.

A P P E N D I X B

(Clause 4.1)

**SAMPLING OF 2-AMINO-5-DIETHYLAMINOTOLUENE
HYDROCHLORIDE, PHOTOGRAPHIC GRADE****B-1. GENERAL REQUIREMENTS OF SAMPLING**

B-1.1 For general requirements of sampling, the methods given in IS : 8883 (Part 1)-1978* may be followed.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of processing shall constitute a lot. If a consignment is declared or known to consist of different batches of processing, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

B-2.1.1 Tests for determining the conformity of the lot to the requirements of the specifications shall be done on each lot separately.

B-2.2 The number of containers to be selected shall depend upon the size of the lot and shall be in accordance with the Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED

LOT SIZE	NUMBER OF CONTAINERS TO BE SELECTED
(1)	(2)
Up to 50	3
51 to 100	4
101 to 150	5
151 and above	7

B-2.3 The containers shall be selected from the lot at random and in order to ensure randomness of selection, the method given in IS : 4905-1968† may be followed.

B-3. NUMBER OF TESTS

B-3.1 Tests for determination of 'free base' percent by mass and 'hydrochloric acid', percent by mass shall be determined on individual containers and for the remaining other characteristics tests shall be conducted on composite sample.

*Methods of sampling chemicals and chemical products: Part I General requirements and precautions.

†Methods for random sampling.

B-4. CRITERIA FOR CONFORMITY

B-4.1 For all those characteristics for which individual tests have been conducted, average (\bar{X}) and range (\bar{R}) shall be calculated, range being the difference between the maximum and minimum of the test results and

$$\text{Average} = \frac{\text{Sum of the test results}}{\text{Number of tests}}$$

For free, base percent by mass and hydrochloric acid percent by mass, the lot shall be declared as conforming to the specification if:

$$R/U - L \leq 0.9,$$

$$\bar{X} + 0.6 R \leq U, \text{ and}$$

$$\bar{X} - 0.6 R \geq L.$$

where U is upper specification limit and L is the lower specification limit.

B-4.2 For composite sample, the lot shall be declared as conforming to the requirements of this specification if none of the test fails.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 kg.m/s ²
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m ²
Frequency	hertz	Hz	1 Hz = 1 c/s (s ⁻¹)
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m ²